Federal Democratic Republic of Ethiopia

EDICT OF GOVERNMENT

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ET ISO 15753 (2012) (English): Animal vegetables fats and oils -- Determination of polycyclic aromatic hydrocarbons

ISO INSIDE

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ETHIOPIAN STANDARD

ES ISO 15753:2012

First edition

Exclusion of olive pomace oil from the scope

(Identical with ISO 15753:2006)

ICS: 67.200.10

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Foreword

This Ethiopian Standard has been prepared under the direction of the Technical Committee for Animal and vegetables fats and oils (TC 23) and published by the Ethiopian Standards Agency (ESA).

The standard is identical with ISO 15753:2006, Exclusion of olive pomace oil from the scope, published by International Organization for Standardization (ISO).

For the purpose of this Ethiopian Standard, the adopted text shall be modified as follows.

- The phrase “International Standard” shall be read as “Ethiopian Standard”; and
- A full stop (.) shall substitute comma (,) as decimal marker.

Animal and vegetable fats and oils — Determination of polycyclic aromatic hydrocarbons

1 Scope

This International Standard describes two methods for the determination of 15 polycyclic aromatic hydrocarbons (PAHs) in animal and vegetable fats and oils:

- a general method, and
- a method specific for coconut oil and vegetable oils with short-chain fatty acids.

These methods are not quantitative for the very volatile compounds such as naphthalene, acenaphthene and fluorene. Due to interferences provided by the matrix itself, palm oil and olive pomace oil cannot be analysed using this method.

The quantification limit is 0,2 µg/kg for almost all compounds analysed, except for fluoranthene and benzo(*g*,*h*,*i*)perylene where the quantification limit is 0,3 µg/kg, and indeno(1,2,3-*c*,*d*)pyrene where the quantification limit is 1 µg/kg.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 661, *Animal and vegetable fats and oils — Preparation of test sample*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
polycyclic aromatic hydrocarbon
PAH
compound that contains two or more condensed (fused) aromatic hydrocarbon rings and the content of which can be determined according to the method specified in this International Standard

NOTE 1 The content is given in micrograms per kilogram.

NOTE 2 In general PAHs are divided into light PAHs with two to four aromatic rings, and heavy PAHs with five or more aromatic rings.

EXAMPLES

Light PAHs include:
naphthalene (CAS RN [91-20-3]), acenaphthene (CAS RN [83-32-9]), acenaphthylene (CAS RN [208-96-8]), fluorene (CAS RN [86-73-7]), anthracene (CAS RN [120-12-7]), phenanthrene (CAS RN [85-01-8]), fluoranthene (CAS RN [206-44-0]), chrysene (CAS RN [218-01-9]), benz(*a*)anthracene (CAS RN [56-55-3]), pyrene (CAS RN [129-00-0]).

Heavy PAHs include:
benzo(*a*)pyrene (CAS RN [50-32-8]), benzo(*b*)fluoranthene (CAS RN [205-99-2]), benzo(*k*)fluoranthene (CAS RN [207-08-9]), benzo(*g*,*h*,*i*)perylene (CAS RN [191-24-2]), dibenz(*a*,*h*)anthracene (CAS RN [53-70-3]), indeno(1,2,3-*c*,*d*)pyrene (CAS RN [193-39-5]).

4 Principle

The polycyclic aromatic hydrocarbons are extracted with an acetonitrile/acetone mixture followed by purification on C18 reversed-phase and then Florisil bonded-phase cartridges. Determination of the content of the individual polycyclic aromatic hydrocarbons after separation is achieved by means of high-pressure liquid chromatography (HPLC) and by measuring the fluorescence at various excitation and emission wavelengths.

5 Reagents and materials

WARNING — Attention is drawn to the regulations governing the handling of dangerous matter. Technical, organizational and personal safety measures must be followed.

Use only reagents of recognized analytical grade unless otherwise stated.

Check the quality of solvents before use by concentrating the solvent about 1 000 times by evaporation and analysing the concentrate by HPLC (300 ml to 300 µl). The chromatogram shall be free from peaks in the elution area of PAHs.

5.1 **Methanol**, ‘ultra resi-analysed’ grade[1].

5.2 **Hexane**, HPLC grade[1].

5.3 **Acetonitrile**, HPLC grade[1].

5.4 **Acetone**, HPLC grade[1].

5.5 **Dichloromethane**, HPLC grade[1].

5.6 **Toluene**, HPLC grade[1].

5.7 **Water**, HPLC grade[1].

5.8 **Tetrahydrofuran**, HPLC grade[1].

5.9 **Solvent mixture 1**: acetonitrile/acetone (volume fraction 60 % / 40 %).

Quantity used per sample: 41 ml for general method, 36 ml for method specific for coconut oil.

5.10 **Solvent mixture 2**: acetonitrile/acetone (volume fraction 80 % / 20 %).

Quantity used per sample: 2 × 11 ml for method specific for coconut oil.

5.11 **Solvent mixture 3**: hexane/dichloromethane (volume fraction 75 % / 25 %).

Quantity used per sample: 7 ml for general method, 2 × 7 ml for method specific for coconut oil.

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[1] These can be obtained from, for example, Baker.

This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products. Equivalent products may be used if they can be shown to lead to the same results.

5.12 Mixture of tetrahydrofuran/methanol (volume fraction 50 % / 50 %).

5.13 Standard solution with 16 certified EPA Priority PAHs in toluene[2], at a concentration of 100 µg/ml (100 mg/l): naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benz(*a*)anthracene, chrysene, benzo(*b*)fluoranthene, benzo(*k*)fluoranthene, benzo(*a*)pyrene, dibenz(*a*,*h*)anthracene, benzo(*g*,*h*,*i*)perylene, indeno(1,2,3-*c*,*d*)pyrene. To be stored at −20 °C.

Before use, allow the solution to warm up to ambient temperature for at least 1 h.

NOTE Acenaphthylene is not fluorescent and, thus, it cannot be determined by these methods.

5.14 Stock standard solution, 200 ng/ml (200 µg/l).

Add 100 µl of standard solution (5.13) with a 250 µl syringe (6.11) to a 50 ml volumetric flask (6.20) and dilute to the mark with acetonitrile.

5.15 Working standard solution, 50 ng/ml (50 µg/l).

Add 250 µl of stock standard solution (5.14) with a 250 µl syringe (6.11) to 750 µl of THF/methanol mixture (5.12) or acetonitrile (5.3).

5.16 C18 bonded-phase cartridges[3], 2 g phase, 12 ml capacity.

5.17 Florisil bonded-phase cartridges[3], 500 mg phase, 3 ml capacity.

5.18 Stream of nitrogen, pressure regulated at 34,5 kPa (5 psi, about 1,5 l/min).

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

The use of disposable glass tubes is acceptable. The general use of glass is necessary as plastics can contain PAHs.

6.1 Centrifuge, capable of attaining at least 4 000 min^{-1}, suitable for 100 ml and 10 ml tubes.

6.2 HPLC system with binary gradient elution, with solvent reservoir of 1 l capacity, a mobile phase liner filter, pump, autosampler, column temperature regulation set at 25 °C, fluorescence detector programmable over time for various excitation and emission wavelengths, and computer-assisted acquisition and data treatment.

6.3 C18 reversed-phase column[4], 250 mm in length, 4,6 mm internal diameter, 5 µm particles, suitable for PAH analysis.

6.4 Vortex mixer.

6.5 Automatic evaporator[5], for 10 ml tube (optional), or water bath (6.6).

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[2] This can be obtained from, for example, Promochem.

[3] This can be obtained from, for example, Varian.

This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products. Equivalent products may be used if they can be shown to lead to the same results.

[4] This can be obtained from, for example, Vydac, ref. 201TP54.

[5] This can be obtained from, for example, Zymark, Zymark TurboVap LV evaporator.

Recommended operating conditions:

— temperature of water bath	35 °C;
— nitrogen pressure	34,5 kPa.

6.6 Water bath, regulated at 35 °C.

6.7 Balance, with readability of 0,1 mg.

6.8 Centrifuge tubes, of 100 ml capacity (one per sample).

6.9 Conical centrifuge tubes, of 11 ml capacity (three per sample), with PTFE septa and closed top screw caps (one per sample).

6.10 Graduated cylinders.

6.11 Microsyringe, 250 µl.

6.12 Syringe, 1 000 µl.

6.13 Graduated pipette, 5 ml.

6.14 Syringe, 5 ml, equipped with an adapter cap for SPE cartridges.

6.15 Vials for autosampler.

6.16 Microvials, of 250 µl capacity, adapted for HPLC system.

6.17 Ultrasonic bath, with water temperature not higher than 40 °C.

6.18 Pasteur pipettes, with cotton wool in the top part to prevent contamination.

6.19 Device composed of stand and pincers6), to hold SPE cartridges or, if available, an automatic SPE work station.

NOTE Depending on the SPE sample processing station used, the proposed extraction methods may require slight adaptations (times, pressure, volumes).

6.20 Volumetric flask, of capacity 50 ml.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5555.

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6) This can be obtained from, for example, Zymark, Zymark Rapid Trace.

This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products. Equivalent products may be used if they can be shown to lead to the same results.

8 Sample preparation

Prepare the test sample in accordance with the method given in ISO 661. Before sampling, the liquid samples shall be at room temperature and homogenized by magnetic agitation.

Sample the solid matrix by melting the entire sample or by melting and homogenizing several core samples.

9 Procedure for determination of PAHs from fats and oils: General method

9.1 Preliminary remarks

In order to obtain repeatable results, the ambient temperature of the laboratory shall be regulated (⩽ 20 °C). This is a very important condition for the extraction of PAHs from coconut oil (or vegetable oils containing short-chain fatty acids). These oils contain fatty acids with short and long chains; when the ambient temperature is higher than 20 °C, the solubility of short-chain fatty acids increases.

Before use, rinse the whole vessel three times with hexane (5.2).

Each sequence of samples shall include a blank (9.2), and a standard solution extracted under the same conditions as the sample in order to calculate the recovery values of the extraction (9.3). The recovery values shall be within the range 70 % to 110 %. The mean recovery values are given in Table A.1.

For a quantitative analysis, two test portions shall be extracted and analysed separately, the final result being the mean value of the results of these two subsamples.

It is not possible to complete the entire analysis within a single day. Sample extracts shall be stored overnight under deep-freeze conditions of at least −18 °C:

- 1st day: step 1, step 2 and step 3, up to purification on C18 cartridge (see Figure A.1).
- 2nd day: step 3, purification on Florisil cartridge and preparation of HPLC system for sample analysis (see Figure A.1).
- following night and day(s): analysis of the samples (see Table A.2).

9.2 Blank

To ensure the absence of contamination of solvents and cartridges, the purification procedure (according to 9.5, 9.6 and Clause 11) shall first be carried out on a blank sample (sample with solvent mixture but with the oil omitted). The chromatogram obtained shall be free from the compounds of interest. If the chromatogram contains interferences, the source of interferences shall be determined and eliminated. Blank values cannot be used to correct sample values as blank values are generally not homogenous (repeatability).

9.3 Determination of recovery values (without matrix)

In order to verify the extraction efficiency of cartridges, carry out a test with a standard solution. Spike 1 750 µl of solvent mixture 1 (5.9) with 250 µl of working standard solution (5.15) with a 250 µl syringe (6.11). Transfer to a C18 cartridge and treat as described in 9.5, 9.6 and Clause 11).

WARNING — When removing solvents under a stream of nitrogen (see 9.5.6), do not evaporate to dryness but leave about 50 µl in the vial, otherwise volatile PAHs will be lost.

9.4 **Extraction** (liquid/liquid extraction)

9.4.1 The flow chart of the isolation procedure is given in Figure A.1.

9.4.2 Weigh, to the nearest 1 mg, about 2,5 g of the sample into a 100 ml centrifuge tube (6.8). Add 10 ml of solvent mixture 1 (5.9).

9.4.3 Agitate the centrifuge tube for 30 s with the vortex mixer (half speed), and then put the tube in an ultrasonic bath (6.17) for 5 min.

9.4.4 Centrifuge for 5 min at 4 000 min^{-1}.

9.4.5 Carefully remove the top layer with a Pasteur pipette (6.18) and transfer it to a weighed conical tube (6.9).

9.4.6 Evaporate the solvent from the conical tube for 30 min to 40 min, under a stream of nitrogen (5.18), using either a water bath at 35 °C (6.6) or an automatic evaporator (6.5).

9.4.7 Repeat the extraction twice with a further 10 ml of solvent mixture 1 (5.9). Concentrate the extracts in the same conical tube under a stream of nitrogen (5.18) using water bath set at 35 °C (6.6) or using an automatic evaporator (6.5). The fat residue should be about 200 mg to 800 mg.

If the fat residue mass is higher than 800 mg, then the general method (Clause 9) is not suitable and the method specific for coconut oil should be used (Clause 10).

9.5 **Purification on C18-bonded phase cartridge** (solid/liquid extraction)

9.5.1 Cartridge conditioning: Put the cartridge (5.16) on a stand (6.19). Rinse the cartridge with 2 volumes of 12 ml of methanol (5.1) then 2 volumes of 12 ml of acetonitrile (5.3). Allow the solvent to flow through under atmospheric pressure.

9.5.2 Put a weighed conical tube (6.9) under the cartridge (5.16).

9.5.3 With a syringe (6.12) or a graduated pipette (6.13), introduce 2 ml of solvent mixture 1 (5.9) into the conical tube containing residual fat material (9.4.6). Agitate the tube with the vortex mixer (6.4) for 15 s. Centrifuge for 30 s. Transfer the top layer to the cartridge (5.16) with a Pasteur pipette (6.18). Repeat the operation twice (2 ml of solvent mixture 1, mixing, centrifuging and transferring onto the cartridge). Collect the solvent eluting from the cartridge together with the elution solvent.

9.5.4 Add 5 ml of solvent mixture 1 (5.9) to the top of the cartridge (5.16) and allow the elution to proceed under atmospheric pressure.

9.5.5 Using a syringe (6.14), inject air into the cartridge in order to elute the remaining solvent and any PAHs which could be retained in the phase.

9.5.6 Remove solvents under a stream of nitrogen (5.18) using a water bath set at 35 °C (6.6) or an automatic evaporator (6.5). The fat residue should be not more than 50 mg.

9.5.7 Dilute the residue in 1 ml of hexane (5.2), measured with a syringe (6.12). Close the conical tube hermetically and store at −18 °C until further use.

9.6 **Purification on Florisil-bonded phase cartridge** (solid/liquid extraction)

9.6.1 Allow the extract (9.5.7) to warm up to ambient temperature for at least 1 h.

9.6.2 Cartridge conditioning: Put the cartridge (5.17) on a stand (6.19). Rinse the cartridge with 5 volumes of 3 ml of dichloromethane (5.5) then 4 volumes of 3 ml of hexane (5.2).

9.6.3 Put a weighed conical tube (6.9) under the cartridge (5.17).

9.6.4 Transfer the extract (9.5.7) to the cartridge (5.17) with a Pasteur pipette (6.18).

9.6.5 Introduce 1 ml of solvent mixture 3 (5.11), using a syringe (6.12) or a graduated pipette (6.13), to the conical tube containing the extract. Agitate it for 15 s with the vortex mixer and transfer it to the cartridge (5.17). Rinse the tube with 2 × 2 ml of solvent mixture 3 (5.11) and transfer it onto the cartridge. Collect the solvent eluting from the cartridge together with the elution solvent.

Pay careful attention to avoid contact between the pipette and the conical tube in order to prevent cross contamination.

9.6.6 Elute 4 ml of solvent mixture 3 (5.11) through the cartridge (5.17). Using a syringe (6.14), inject air into the cartridge in order to elute the remaining solvent.

9.6.7 Concentrate the solution under a stream of nitrogen (5.18), using a water bath set at 35 °C (6.6) or an automatic evaporator (6.5), to about 1 ml (takes 10 min to 15 min). Add about 0,5 ml of toluene (5.6) (keeper) and continue to evaporate until about 50 µl remain.

The solvent should not be removed completely.

9.6.8 The exact volume is determined by weighing the conical tube, and calculating using the density of toluene. Add the necessary volume of solvent [MeOH/THF (5.12), or acetonitrile (5.3)], V_{add}, to make up to 250 µl:

$$V_{\text{add}} = 250 - \frac{m}{d}$$

where

m is the sample mass, in milligrams;

d is the density of toluene (0,866 9 kg/m^3).

9.6.9 Transfer the sample to a microvial (6.16) placed in a vial (6.15).

10 Procedure for determination of PAHs from fats and oils: Method specific for coconut oil

10.1 **First extraction** (liquid/liquid extraction)

10.1.1 The flow chart of the isolation procedure is given in Figure A.2.

10.1.2 Weigh, to the nearest 1 mg, about 2 g of the sample into a 100 ml centrifuge tube (6.8). Add 10 ml of solvent mixture 1 (5.9).

10.1.3 Agitate the centrifuge tube for 30 s with the vortex mixer (half speed), and then put the tube in an ultrasonic bath (6.17) for 5 min.

10.1.4 Centrifuge for 5 min at 4 000 min^{-1}.

10.1.5 Carefully remove the top layer with a Pasteur pipette (6.18) and introduce it into a conical tube (6.9).

10.1.6 Evaporate the solvent from the conical tube for 30 min to 40 min under a stream of nitrogen (5.18) using the water bath set at 35 °C (6.6) or an automatic evaporator (6.5).

10.1.7 Repeat the extraction twice, with a further 10 ml of solvent mixture 1 (5.9). Concentrate the extracts in the same conical tube under a stream of nitrogen (5.18) using water bath set at 35 °C (6.6) or an automatic evaporator (6.5).

10.2 Second extraction (liquid/liquid extraction)

10.2.1 With a syringe (6.12) or a graduated pipette (6.13), introduce 2 ml of solvent mixture 1 (5.9) to the conical tube containing residual fat material (10.1.7). Agitate the tube with the vortex mixer for 15 s. Centrifuge for 30 s. Divide the extract into two equal parts in two weighed conical tubes (6.9).

Pay careful attention to avoid contact between the pipette and the conical tube in order to prevent cross contamination.

10.2.2 Repeat twice the procedure detailed in 10.2.1 using the same conical tubes.

10.2.3 Concentrate the two extracts under a stream of nitrogen (5.18) using the water bath set at 35 °C (6.6) or an automatic evaporator (6.5). Each fat residue should be about 250 mg.

10.3 Purification on C18-bonded phase cartridge (solid/liquid extraction)

10.3.1 Cartridge conditioning: Put two cartridges (5.16) on a stand (6.19). Rinse the cartridges with 2 volumes of 12 ml of methanol (5.1) then 2 volumes of 12 ml of acetonitrile (5.3). Allow the solvent to flow under atmospheric pressure.

10.3.2 Put a weighed conical tube (6.9) under each cartridge (5.16).

10.3.3 With a syringe (6.12) or a graduated pipette (6.13), introduce 2 ml of solvent mixture 2 (5.10) to the two conical tubes containing the residual fat material (10.2.3). Agitate the tubes with the vortex mixer for 15 s. Transfer the whole extract of each conical tube to each cartridge (5.16) with a Pasteur pipette (6.18). Rinse the tube with 2 × 2 ml of solvent mixture 2 (5.10) and transfer it onto the cartridge. Collect the solvent eluting from the cartridge together with the elution solvent.

10.3.4 Elute 5 ml of solvent mixture 2 (5.10) through the cartridges (**do not** inject air on the cartridges).

10.3.5 Remove solvents under a stream of nitrogen (5.18) using the water bath set at 35 °C (6.6) or an automatic evaporator (6.5). The fat residue should be about 50 mg to 100 mg.

10.3.6 Dilute the residues in 1 ml of hexane (5.2), measured with a syringe (6.12). Close the conical tube hermetically and store at –18 °C until the following day.

10.4 Purification on Florisil-bonded phase cartridge (solid/liquid extraction)

10.4.1 Allow the extracts (10.3.6) to warm up to ambient temperature for at least 1 h.

10.4.2 Cartridge conditioning: Put two cartridges (5.17) on a stand (6.19). Rinse the cartridges with 5 volumes of 3 ml of dichloromethane (5.5) then 4 volumes of 3 ml of hexane (5.2).

10.4.3 Put a weighed conical tube (6.9) under each cartridge (5.17).

10.4.4 Transfer each extract (10.3.6) to each cartridge (5.17) with a Pasteur pipette (6.18).

10.4.5 Introduce 1 ml of solvent mixture 3 (5.11), using a syringe (6.12) or graduated pipette (6.13), to each extract tube. Agitate it for 15 s with the vortex mixer and transfer to the cartridge (5.17). Rinse the tube with 2 × 2 ml of solvent mixture 3 (5.11) and transfer it onto the cartridge. Collect the solvent eluting from the cartridge together with the elution solvent.

10.4.6 Elute 4 ml of solvent mixture 3 (5.11) through the cartridges (5.17) (**do not** inject air into the cartridge).

10.4.7 Concentrate the solution under a stream of nitrogen (5.18), using the water bath set at 35 °C (6.6) or an automatic evaporator (6.5), to about 1 ml (takes 10 min to 15 min). Collect the two extracts in the same conical tube by transferring the extract of one conical tube into the other one. Rinse the empty conical tube with 3 × 1 ml of hexane (5.2). Add about 0,5 ml of toluene (5.6) (keeper) using a syringe (6.12) and continue to evaporate to about 50 µl.

The solvent should not be removed completely.

10.4.8 The exact volume is determined by weighing the conical tube and calculating using the density of toluene. Add the necessary volume of solvent [MeOH/THF (5.12) or acetonitrile (5.3)], V_{add}, to make up to 200 µl:

$$V_{\text{add}} = 200 - \frac{m}{d}$$

where

m is the mass sample, in milligrams;

d is the density of toluene (0,866 9 kg/m^3).

10.4.9 Transfer the sample to a microvial (6.16) placed in a vial (6.15).

11 High-performance liquid chromatography (HPLC)

11.1 Operating conditions

Mobile phase: Solvent mixture A: acetonitrile
Solvent mixture B: acetonitrile/water (50:50 volume fraction)

Flow: 1,2 ml/min
Injector volume: 20 µl
Column temperature: 25 °C

An example of the programming of solvents is given as an example in Table 1. The gradient elution programme should be changed according to the column being used.

Table 1 — Gradient elution programme on C18 reversed-phase column

Time min	Solvent mixture A %	Solvent mixture B %
0	0	100
5	0	100
27	60	40
36	100	0
41	100	0
43	0	100
45	0	100

11.2 Detection parameters

Excitation and emission spectra can vary slightly with different instruments. In order to determine the maximum excitation and emission wavelengths, the spectrum of each compound shall be obtained using a working standard solution of 16 PAHs (5.15), as follows.

- First, determine the maximum emission wavelength by scanning emission between 300 nm to 550 nm at an arbitrary excitation wavelength (see Table 2).

- Then determine the maximum excitation wavelength by scanning excitation between 200 nm to 350 nm and recording at the emission wavelength determined previously.

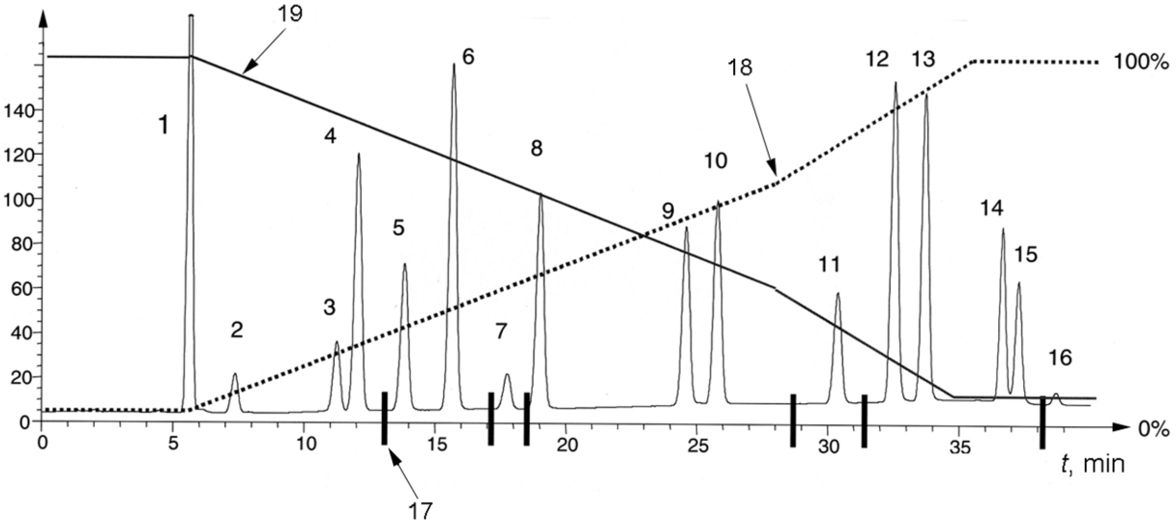
For each of the seven groups of compounds (see Table 2) having close retention times, choose a set of excitation/emission wavelengths as near as possible to the maximum wavelength of each compound.

As an example, a choice of wavelengths obtained with a HP 1100 fluorimeter is given in Table 2.

Table 2 — Detection programme

Group	Component	Time min	Excitation wavelength nm	Emission wavelength nm
1	Naphthalene Acenaphthene Fluorene	0	270	324
2	Phenanthrene Anthracene	12,6	248	375
3	Fluoranthene	16,4	280	462
4	Pyrene Benz(*a*)anthracene Chrysene	18,05	270	385
5	Benzo(*b*)fluoranthene	28,0	256	446
6	Benzo(*k*)fluoranthene Benzo(*a*)pyrene Dibenz(*a*,*h*)anthracene Benzo(*g*,*h*,*i*)perylene	31,1	292	410
7	Indeno(1,2,3-*c*,*d*)pyrene	38,0	274	507

Under these conditions, the chromatogram in Figure 1 is obtained for a standard solution of 16 PAHs.



Key

1	Toluene
2	Naphthalene
3	Acenaphthene
4	Fluorene
5	Phenanthrene
6	Anthracene
7	Fluoranthene
8	Pyrene
9	Benz(*a*)anthracene
10	Chrysene
11	Benzo(*b*)fluoranthene
12	Benzo(*k*)fluoranthene
13	Benzo(*a*)pyrene
14	Dibenz(*a*,*h*)anthracene
15	Benzo(*g*,*h*,*i*)perylene
16	Indeno(1,2,3-*c*,*d*)pyrene
17	change of wavelength
18	solvent mixture A (acetonitrile)
19	solvent mixture B (acetonitrile/water 50:50, volume fraction)

Figure 1 — Chromatogram of working standard solution (5.15)

11.3 Analysis of samples and standards

Samples and standards shall be injected at least twice. The relative standard deviation of the area for two injections of the same sample should not exceed 5 %. If the standard deviation exceeds 5 %, HPLC conditions should be optimized and samples and standards should be injected a second time.

The sequence of injections should be as follows:

a) standard solution (5.15);

b) standard solution extracted (see 9.3);

c) samples.

As an example, a detailed sequence of injection is given in Table A.2.

The presence of PAHs in the samples is determined by comparing the retention time of the sample chromatograms with the retention time of the nearest reference sample chromatogram. The chromatogram presented in Figure A.3 is obtained for a virgin olive oil sample.

11.4 Confirmation of the presence of PAHs

In addition, inject each sample again twice: one for recording the emission spectrum of each compound and the other one for recording the excitation spectrum. This enables confirmation of the presence of the compound of interest by comparing its spectra with the reference spectra.

12 Expression of results

Before the calculation, the following points shall be checked:

- the quality of the blank;
- the relative standard deviation between two injections of the same sample (should not exceed 5 %);
- the recovery percentage of each PAH (9.3 and Table A.1).

The calculation is achieved by external calibration.

Calculation of the content c_i of a PAH in the sample, in micrograms per kilogram:

$$c_i = \frac{A_i \times c_{i\mathrm{r}} \times V}{A_{i\mathrm{r}} \times m}$$

where

A_i is the peak area (mean of two injections) of the respective PAH in the sample solution;

$A_{i\mathrm{r}}$ is the peak area (mean of two injections) of the respective PAH in the working standard solution (5.15);

$c_{i\mathrm{r}}$ is the concentration of the respective PAH in the working standard solution (5.15), in micrograms per kilogram;

V is the volume of the final extract, in millilitres;

m is the mass of sample, in grams.

For quantitative analysis, each sample shall be extracted twice. The content of each PAH is given by the mean of the two values obtained, expressed to the nearest 0,1 µg/kg.

13 Precision

13.1 Interlaboratory test

The results of an interlaboratory test of the precision of the method are given in Annex B.

The values for repeatability and reproducibility limits are expressed for the 95 % probability level and may not be applicable to concentration ranges and matrices other than those given.

13.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases exceed the repeatability limit r calculated from the following:

Individual PAH concentration, c (µg/kg)	0 µg/kg to 10 µg/kg	10 µg/kg to 100 µg/kg
Repeatability limit, r (µg/kg)	$0{,}37 \times c$	$0{,}12 \times c + 3{,}7$

where c is the mean of the two test results, expressed in micrograms per kilogram.

13.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases exceed the reproducibility limit R calculated from the following:

Individual PAH concentration, c (µg/kg)	0 µg/kg to 10 µg/kg	10 µg/kg to 40 µg/kg	40 µg/kg to 100 µg/kg
Reproducibility limit, R (µg/kg)	$1{,}1 \times c$	$0{,}86 \times c + 1{,}5$	40 µg/kg

where c is the mean of the two test results, expressed in micrograms per kilogram.

14 Test report

The test report shall specify:

a) all information necessary for the complete identification of the sample;

b) the test method used, with reference to this International Standard;

c) all operating details not specified in this method, or regarded as optional, together with any incidents which may have influenced the results;

d) the test results and the units in which they are expressed;

e) the recovery values for each PAH.

Annex A
(informative)

Recovery values, flow charts, chromatograms and injection sequences

Table A.1 — Mean recovery values of polycyclic hydrocarbons in oils

Compound	General method	Coconut oil
Phenanthrene	> 70 %	> 70 %
Anthracene	> 70 %	> 70 %
Fluoranthene	> 70 %	> 70 %
Pyrene	> 70 %	> 70 %
Benz(*a*)anthracene	> 70 %	> 70 %
Chrysene	> 70 %	> 70 %
Benzo(*b*)fluoranthene	> 70 %	> 70 %
Benzo(*k*)fluoranthene	> 70 %	> 70 %
Benzo(*a*)pyrene	> 70 %	> 60 %
Dibenzo(*a*,*h*)anthracene	> 70 %	> 60 %
Benzo(*g*,*h*,*i*)perylene	> 60 %	> 50 %
Indeno(1,2,3-*c*,*d*)pyrene	> 70 %	> 50 %

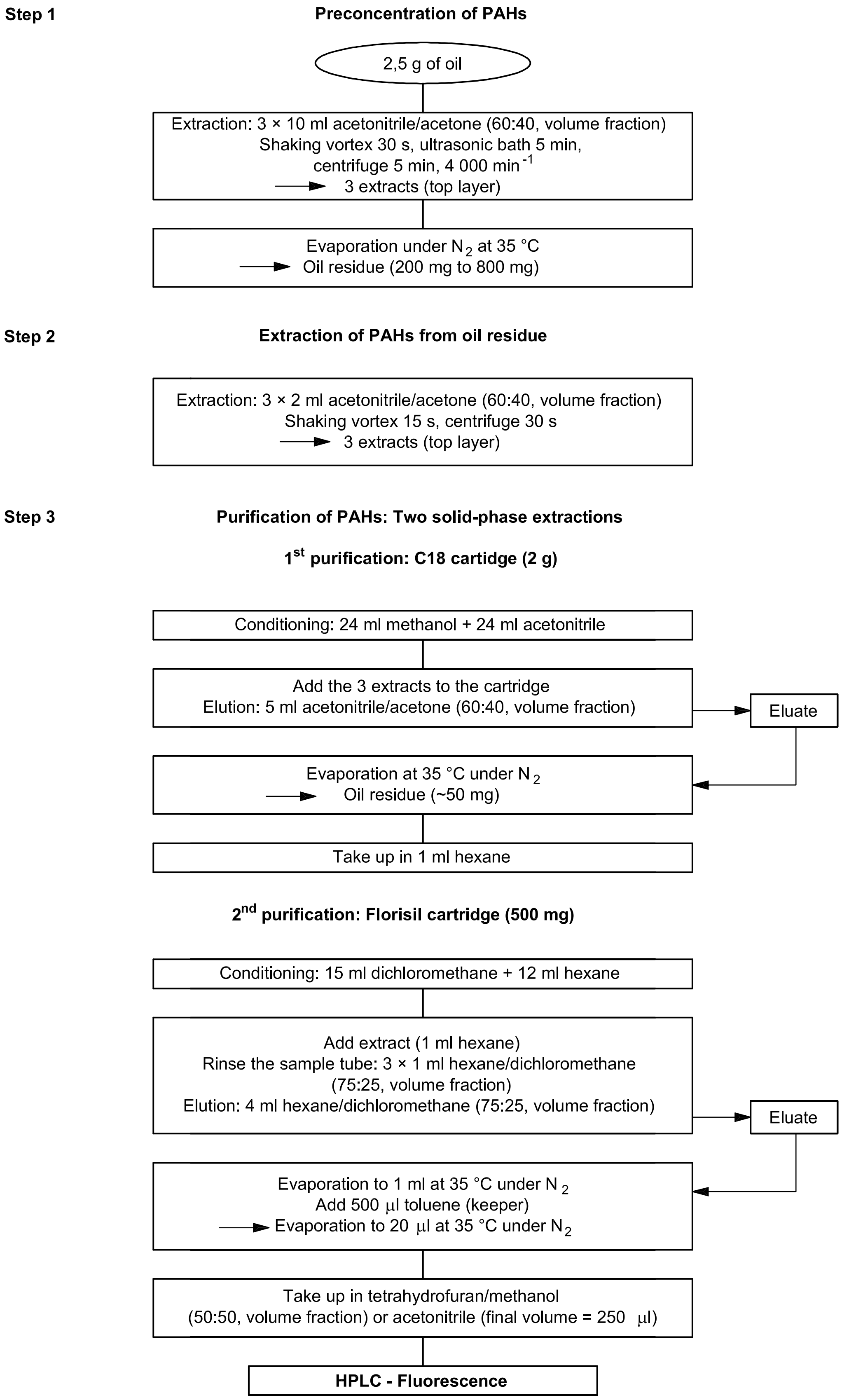

Step 1
Preconcentration of PAHs
2,5 g of oil
Extraction: 3 × 10 ml acetonitrile/acetone (60:40, volume fraction)
Shaking vortex 30 s, ultrasonic bath 5 min,
centrifuge 5 min, 4 000 min^{-1}
→ 3 extracts (top layer)
Evaporation under N_2 at 35 °C
→ Oil residue (200 mg to 800 mg)
Step 2
Extraction of PAHs from oil residue
Extraction: 3 × 2 ml acetonitrile/acetone (60:40, volume fraction)
Shaking vortex 15 s, centrifuge 30 s
→ 3 extracts (top layer)
Step 3
Purification of PAHs: Two solid-phase extractions
1st purification: C18 cartridge (2 g)
Conditioning: 24 ml methanol + 24 ml acetonitrile
Add the 3 extracts to the cartridge
Elution: 5 ml acetonitrile/acetone (60:40, volume fraction)
Eluate
Evaporation at 35 °C under N_2
→ Oil residue (~50 mg)
Take up in 1 ml hexane
2nd purification: Florisil cartridge (500 mg)
Conditioning: 15 ml dichloromethane + 12 ml hexane
Add extract (1 ml hexane)
Rinse the sample tube: 3 × 1 ml hexane/dichloromethane
(75:25, volume fraction)
Elution: 4 ml hexane/dichloromethane (75:25, volume fraction)
Eluate
Evaporation to 1 ml at 35 °C under N_2
Add 500 µl toluene (keeper)
→ Evaporation to 20 µl at 35 °C under N_2
Take up in tetrahydrofuran/methanol
(50:50, volume fraction) or acetonitrile (final volume = 250 µl)
HPLC - Fluorescence

Figure A.1 — Flow chart for the isolation procedure: General method

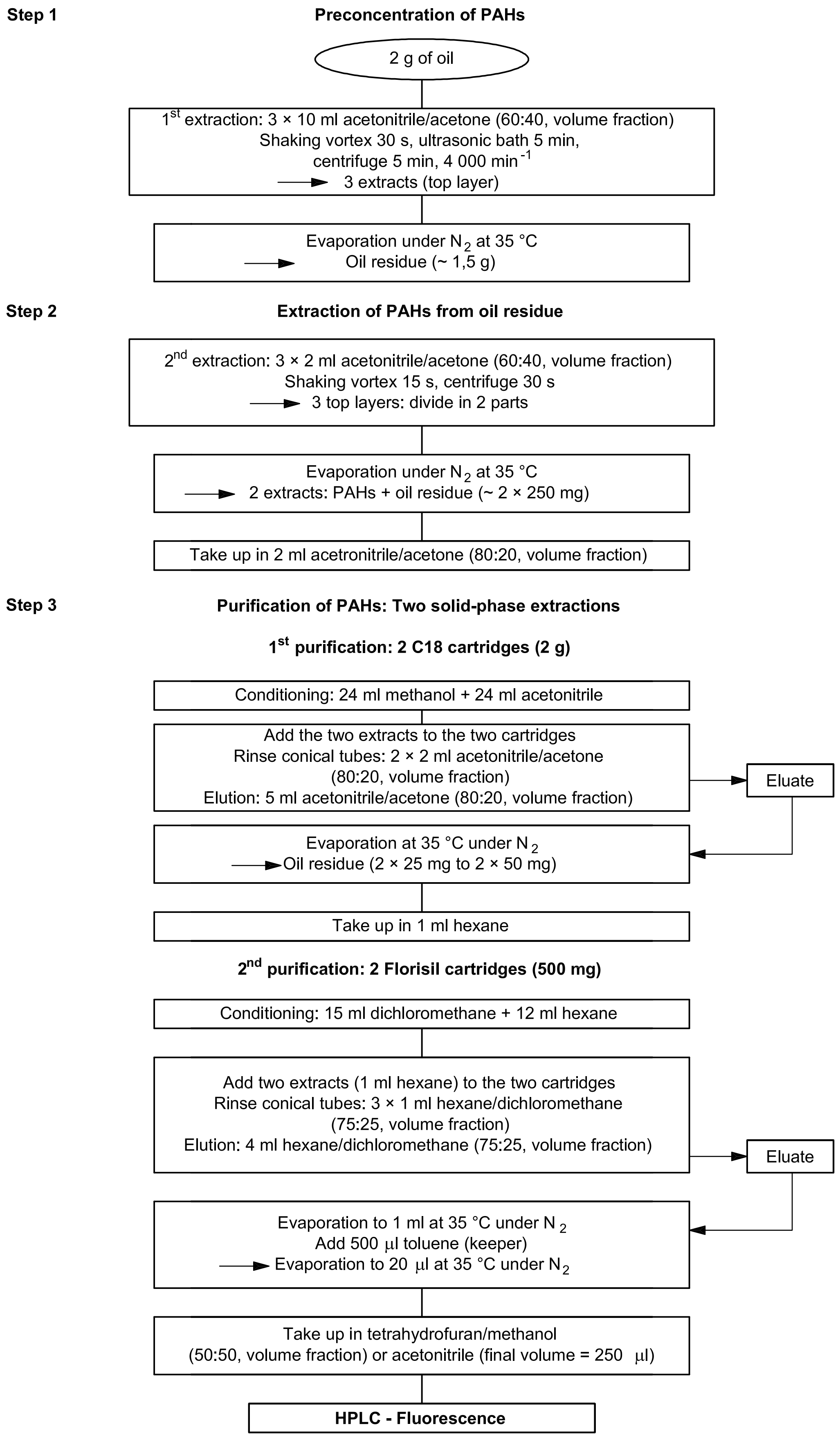
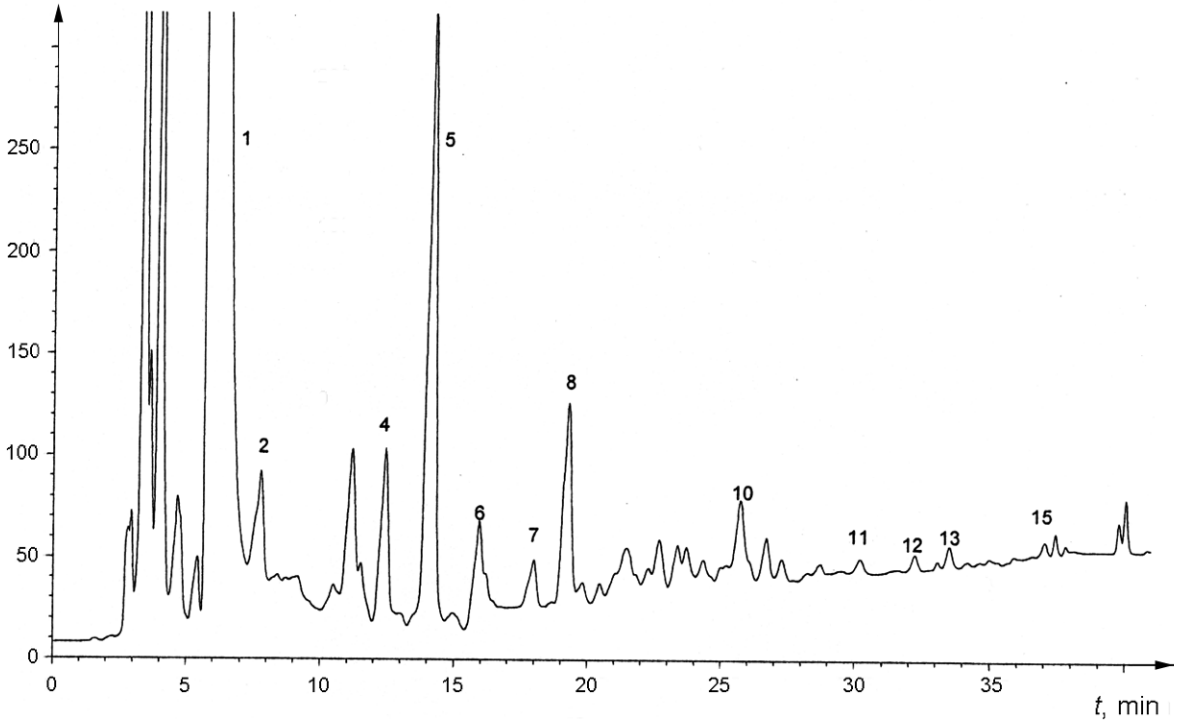

Step 1
Preconcentration of PAHs
2 g of oil
1st extraction: 3 × 10 ml acetonitrile/acetone (60:40, volume fraction)
Shaking vortex 30 s, ultrasonic bath 5 min,
centrifuge 5 min, 4 000 min^{-1}
→ 3 extracts (top layer)
Evaporation under N_2 at 35 °C
→ Oil residue (~ 1,5 g)
Step 2
Extraction of PAHs from oil residue
2nd extraction: 3 × 2 ml acetonitrile/acetone (60:40, volume fraction)
Shaking vortex 15 s, centrifuge 30 s
→ 3 top layers: divide in 2 parts
Evaporation under N_2 at 35 °C
→ 2 extracts: PAHs + oil residue (~ 2 × 250 mg)
Take up in 2 ml acetronitrile/acetone (80:20, volume fraction)
Step 3
Purification of PAHs: Two solid-phase extractions
1st purification: 2 C18 cartridges (2 g)
Conditioning: 24 ml methanol + 24 ml acetonitrile
Add the two extracts to the two cartridges
Rinse conical tubes: 2 × 2 ml acetonitrile/acetone
(80:20, volume fraction)
Elution: 5 ml acetonitrile/acetone (80:20, volume fraction)
Eluate
Evaporation at 35 °C under N_2
→ Oil residue (2 × 25 mg to 2 × 50 mg)
Take up in 1 ml hexane
2nd purification: 2 Florisil cartridges (500 mg)
Conditioning: 15 ml dichloromethane + 12 ml hexane
Add two extracts (1 ml hexane) to the two cartridges
Rinse conical tubes: 3 × 1 ml hexane/dichloromethane
(75:25, volume fraction)
Elution: 4 ml hexane/dichloromethane (75:25, volume fraction)
Eluate
Evaporation to 1 ml at 35 °C under N_2
Add 500 µl toluene (keeper)
→ Evaporation to 20 µl at 35 °C under N_2
Take up in tetrahydrofuran/methanol
(50:50, volume fraction) or acetonitrile (final volume = 250 µl)
HPLC - Fluorescence

Figure A.2 — Flow chart for the isolation procedure: Method specific for coconut oil



Key

1	Toluene	(solvent)	9	Benz(*a*)anthracene	nd
2	Naphthalene	(10,2 µg/kg)	10	Chrysene	(1,0 µg/kg)
3	Acenaphthene	nd	11	Benzo(*b*)fluoranthene	(0,4 µg/kg)
4	Fluorene	(2,2 µg/kg)	12	Benzo(*k*)fluoranthene	(0 2µg/kg)
5	Phenanthrene	(15,2 µg/kg)	13	Benzo(*a*)pyrene	(0,2 µg/kg)
6	Anthracene	(1,4 µg/kg)	14	Dibenz(*a*,*h*)anthracene	nd
7	Fluoranthene	(4,2 µg/kg)	15	Benzo(*g*,*h*,*i*)perylene	(0,4 µg/kg)
8	Pyrene	(4,3 µg/kg)	16	Indeno(1,2,3-*c*,*d*)pyrene	nd

nd = not detected

Figure A.3 — Chromatogram of a virgin olive oil sample

Table A.2 — HPLC injection sequences

Row	Vial	Number of injections	Acquisition method
Determination of content of PAHs			
1	Tetrahydrofuran/methanol (50:50, volume fraction)	1	Specific wavelengths
2	Blank (9.2)	1	Specific wavelengths
3	Standard solution, 50 ng/ml (5.15)	2	Specific wavelengths
4	Standard solution extracted (9.3)	2	Specific wavelengths
5	Sample 1	2	Specific wavelengths
6	Standard solution, 50 ng/ml (5.15)	2	Specific wavelengths
7	Sample 2	2	Specific wavelengths
8	Sample 3	2	Specific wavelengths
9	Standard solution, 50 ng/ml (5.15)	2	Specific wavelengths
10	Sample 4	2	Specific wavelengths
11	Sample 5	2	Specific wavelengths
12	Standard solution, 50 ng/ml (5.15)	2	Specific wavelengths
13	Sample 6	2	Specific wavelengths
14	Sample 7	2	Specific wavelengths
15	Standard solution, 50 ng/ml (5.15)	2	Specific wavelengths
	Total analysis time	23 h	
Confirmation of the presence of PAHs			
1	Sample 1	1	Emission
2	Sample 2	1	Emission
3	Sample 3	1	Emission
4	Sample 4	1	Emission
5	Sample 5	1	Emission
6	Sample 6	1	Emission
7	Sample 7	1	Emission
8	Sample 1	1	Excitation
9	Sample 2	1	Excitation
10	Sample 3	1	Excitation
11	Sample 4	1	Excitation
12	Sample 5	1	Excitation
13	Sample 6	1	Excitation
14	Sample 7	1	Excitation

Annex B
(informative)

Results of the interlaboratory test

An interlaboratory test carried out at the international level in 2002/2003 by ITERG (France), in which 19 laboratories participated, each obtaining two test results for each sample, gave the statistical results (evaluated in accordance with ISO 5725-2) summarized in Table B.1.

Table B.1 — Results of interlaboratory test

Code	PAH	Labs	Mean	s_r	RSD(r)	r	s_R	RSD(R)	R
A	Phenanthrene	15	39,95	4,96	12,4 %	13,89	13,27	33,2 %	37,15
A	Anthracene	15	31,60	3,47	11,0 %	9,71	10,56	33,4 %	29,55
A	Fluoranthene	13	49,70	3,57	7,2 %	9,99	13,68	27,5 %	38,31
A	Pyrene	13	70,36	4,67	6,6 %	13,07	23,47	33,4 %	65,71
A	Benzo[*a*]anthracene	15	30,64	2,21	7,2 %	6,20	9,56	31,2 %	26,78
A	Chrysene	15	34,34	2,39	6,9 %	6,68	10,23	29,8 %	28,63
A	Benzo[*b*]fluoranthene	15	26,00	2,25	8,7 %	6,30	8,61	33,1 %	24,11
A	Benzo[*k*]fluoranthene	14	20,23	1,23	6,1 %	3,44	6,76	33,4 %	18,94
A	Benzo[*a*]pyrene	12	18,19	1,23	6,8 %	3,44	4,44	24,4 %	12,42
A	Dibenzo[*a*,*h*]anthracene	15	18,78	2,46	13,1 %	6,88	7,24	38,5 %	20,27
A	Benzo[*g*,*h*,*i*]perylene	13	11,63	1,56	13,5 %	4,38	4,11	35,3 %	11,51
A	Indeno[1,2,3-*c*,*d*]pyrene	14	12,91	2,88	22,3 %	8,06	5,93	45,9 %	16,61
B	Phenanthrene	17	82,57	4,08	4,9 %	11,42	13,94	16,9 %	39,02
B	Anthracene	17	74,72	3,93	5,3 %	10,99	14,30	19,1 %	40,05
B	Fluoranthene	17	83,18	3,43	4,1 %	9,60	14,23	17,1 %	39,83
B	Pyrene	17	112,81	8,25	7,3 %	23,09	18,82	16,7 %	52,69
B	Benzo[*a*]anthracene	17	70,47	3,75	5,3 %	10,51	11,62	16,5 %	32,54
B	Chrysene	16	80,96	4,77	5,9 %	13,35	13,97	17,3 %	39,11
B	Benzo[*b*]fluoranthene	17	68,95	4,04	5,9 %	11,30	13,32	19,3 %	37,30
B	Benzo[*k*]fluoranthene	17	55,95	2,72	4,9 %	7,62	9,80	17,5 %	27,45
B	Benzo[*a*]pyrene	17	57,78	3,95	6,8 %	11,07	12,61	21,8 %	35,30
B	Dibenzo[*a*,*h*]anthracene	17	48,92	3,46	7,1 %	9,69	14,43	29,5 %	40,40
B	Benzo[*g*,*h*,*i*]perylene	17	41,99	3,05	7,3 %	8,53	16,01	38,1 %	44,82
B	Indeno[1,2,3-*c*,*d*]pyrene	15	45,11	5,77	12,8 %	16,15	18,00	39,9 %	50,39
C	Phenanthrene	17	7,67	0,72	9,4 %	2,01	5,35	69,8 %	14,98
C	Anthracene	15	3,59	0,58	16,1 %	1,62	0,86	23,9 %	2,40
C	Fluoranthene	15	4,52	0,85	18,9 %	2,39	0,98	21,6 %	2,73
C	Pyrene	15	6,12	0,80	13,0 %	2,23	1,19	19,4 %	3,33
C	Benzo[*a*]anthracene	16	3,79	0,53	13,9 %	1,47	0,77	20,4 %	2,16
C	Chrysene	15	4,28	0,64	15,0 %	1,80	0,97	22,6 %	2,71
C	Benzo[*b*]fluoranthene	16	3,58	0,54	15,2 %	1,52	1,00	27,8 %	2,79
C	Benzo[*k*]fluoranthene	14	2,55	0,41	16,2 %	1,16	0,46	18,1 %	1,29
C	Benzo[*a*]pyrene	17	3,21	0,40	12,3 %	1,11	1,35	42,1 %	3,78

Table B.1 (*continued*)

Code	PAH	Labs	Mean	s_r	RSD(r)	r	s_R	RSD(R)	R
C	Dibenzo[*a*,*h*]anthracene	16	2,25	0,36	16,0 %	1,01	1,08	48,1 %	3,03
C	Benzo[*g*,*h*,*i*]perylene	16	1,86	0,32	17,1 %	0,89	0,86	46,3 %	2,41
C	Indeno[1,2,3-*c*,*d*]pyrene	15	1,98	0,34	17,3 %	0,96	1,01	51,2 %	2,84
D	Phenanthrene	18	5,48	1,77	32,3%	4,96	5,07	92,5 %	14,20
D	Anthracene	—	—	—	—	—	—	—	—
D	Fluoranthene	15	0,61	0,18	29,3%	0,50	0,40	65,6 %	1,12
D	Pyrene	16	0,88	0,25	28,8%	0,71	0,61	69,0 %	1,70
D	Benzo[*a*]anthracene	—	—	—	—	—	—	—	—
D	Chrysene	13	0,22	0,04	17,9%	0,11	0,18	81,2 %	0,50
D	Benzo[*b*]fluoranthene	—	—	—	—	—	—	—	—
D	Benzo[*k*]fluoranthene	—	—	—	—	—	—	—	—
D	Benzo[*a*]pyrene	—	—	—	—	—	—	—	—
D	Dibenzo[*a*,*h*]anthracene	13	0,30	0,03	10,7%	0,09	0,37	123,8 %	1,04
D	Benzo[*g*,*h*,*i*]perylene	12	0,27	0,10	35,7%	0,27	0,59	216,9 %	1,64
D	Indeno[1,2,3-*c*,*d*]pyrene	—	—	—	—	—	—	—	—
E	Phenanthrene	16	14,55	1,60	11,0%	4,48	6,09	41,8 %	17,04
E	Anthracene	15	1,08	0,24	22,2%	0,67	0,43	39,7 %	1,20
E	Fluoranthene	15	4,10	0,48	11,6%	1,33	1,19	28,9 %	3,32
E	Pyrene	15	3,69	0,51	13,9%	1,44	1,00	27,1 %	2,80
E	Benzo[*a*]anthracene	15	0,41	0,13	30,5%	0,35	0,40	97,6 %	1,12
E	Chrysene	15	1,17	0,13	11,0%	0,36	0,43	36,6 %	1,20
E	Benzo[*b*]fluoranthene	16	0,40	0,06	16,1%	0,18	0,31	77,7 %	0,87
E	Benzo[*k*]fluoranthene	—	—	—	—	—	—	—	—
E	Benzo[*a*]pyrene	17	0,32	0,04	11,2%	0,10	0,30	94,9 %	0,85
E	Dibenzo[*a*,*h*]anthracene	—	—	—	—	—	—	—	—
E	Benzo[*g*,*h*,*i*]perylene	—	—	—	—	—	—	—	—
E	Indeno[1,2,3-*c*,*d*]pyrene	—	—	—	—	—	—	—	—
F	Phenanthrene	16	5,03	0,76	15,2 %	2,14	5,42	107,7 %	15,17
F	Anthracene	13	0,27	0,06	21,2 %	0,16	0,31	116,4 %	0,88
F	Fluoranthene	13	1,63	0,36	22,1 %	1,01	0,93	57,0 %	2,60
F	Pyrene	17	4,52	0,87	19,2 %	2,43	4,32	95,6 %	12,10
F	Benzo[*a*]anthracene	15	2,50	0,32	12,9 %	0,90	2,79	111,6 %	7,81
F	Chrysene	14	5,36	0,42	7,9 %	1,18	4,86	90,7 %	13,61
F	Benzo[*b*]fluoranthene	11	0,64	0,13	20,1 %	0,36	0,95	149,0 %	2,67
F	Benzo[*k*]fluoranthene	15	1,07	0,20	19,0 %	0,57	1,15	107,8 %	3,23
F	Benzo[*a*]pyrene	12	0,36	0,08	21,8 %	0,22	0,53	145,8 %	1,47
F	Dibenzo[*a*,*h*]anthracene	16	1,26	0,71	56,7 %	2,00	1,62	128,4 %	4,53
F	Benzo[*g*,*h*,*i*]perylene	14	0,78	0,18	22,9 %	0,50	0,97	124,1 %	2,71
F	Indeno[1,2,3-*c*,*d*]pyrene	—	—	—	—	—	—	—	—
G	Phenanthrene	14	3192	258	8,1 %	721	948	29,7 %	2653
G	Anthracene	14	413	58	14,1 %	163	225	54,6 %	631
G	Fluoranthene	13	966	101	10,5 %	284	324	33,5 %	906
G	Pyrene	15	903	86	9,5 %	240	284	31,4 %	794

Table B.1 (*continued*)

Code	PAH	Labs	Mean	s_r	RSD(r)	r	s_R	RSD(R)	R
G	Benzo[*a*]anthracene	14	247	23	9,1 %	63	134	54,2 %	375
G	Chrysene	12	441	16	3,7 %	46	160	36,3 %	448
G	Benzo[*b*]fluoranthene	15	136	11	8,1 %	31	43	31,3 %	119
G	Benzo[*k*]fluoranthene	15	42	5	11,9 %	14	12	28,1 %	33
G	Benzo[*a*]pyrene	15	112	13	11,5 %	36	33	29,7 %	93
G	Dibenzo[*a*,*h*]anthracene	13	15	2	12,8 %	5	12	79,9 %	33
G	Benzo[*g*,*h*,*i*]perylene	14	68	11	16,3 %	31	31	46,2 %	88
G	Indeno[1,2,3-*c*,*d*]pyrene	13	29	6	22,2 %	18	31	108,4 %	88
H	Phenanthrene	15	3,54	0,36	10,2 %	1,01	2,70	76,2 %	7,55
H	Anthracene	15	0,87	0,15	16,8 %	0,41	0,25	29,1 %	0,71
H	Fluoranthene	13	1,10	0,13	12,0 %	0,37	0,30	27,6 %	0,85
H	Pyrene	16	1,69	0,29	16,9 %	0,80	0,90	53,3 %	2,52
H	Benzo[*a*]anthracene	16	0,96	0,11	11,5 %	0,31	0,36	37,6 %	1,01
H	Chrysene	13	0,93	0,11	11,5 %	0,30	0,23	24,2 %	0,63
H	Benzo[*b*]fluoranthene	17	0,95	0,13	13,2 %	0,35	0,40	42,5 %	1,13
H	Benzo[*k*]fluoranthene	17	0,76	0,08	10,3 %	0,22	0,38	50,3 %	1,07
H	Benzo[*a*]pyrene	15	0,64	0,07	10,6 %	0,19	0,16	25,7 %	0,46
H	Dibenzo[*a*,*h*]anthracene	17	0,50	0,08	15,7 %	0,22	0,27	53,6 %	0,75
H	Benzo[*g*,*h*,*i*]perylene	16	0,53	0,11	21,6 %	0,32	0,38	70,8 %	1,05
H	Indeno[1,2,3-*c*,*d*]pyrene	—	—	—	—	—	—	—	—

SAMPLE A: refined coconut oil spiked with a mixture of PAHs at an intermediate level of concentration (~50 µg/kg)

SAMPLE B: virgin olive oil spiked with a mixture of PAHs at a high level of concentration (~100 µg/kg)

SAMPLE C: refined sunflower oil spiked with a mixture of PAHs at a low level of concentration (~5 µg/kg)

SAMPLE D: refined grape seed oil, not spiked

SAMPLE E: virgin olive oil, not spiked

SAMPLE F: refined pomace olive oil, not spiked

SAMPLE G: crude pomace olive oil, not spiked

SAMPLE H: refined sunflower oil spiked with a mixture of PAHs at a low level of concentration (~1 µg/kg)

Labs: Number of laboratories retained after eliminating outliers

Mean: Mean value (µg/kg sample)

s_r: Repeatability standard deviation (µg/kg)

s_R: Reproducibility standard deviation (µg/kg)

RSD(r): Repeatability relative standard deviation (%)

RSD(R): Reproducibility relative standard deviation (%)

r: Repeatability limit ($2{,}8 \times s_r$)

R: Reproducibility limit ($2{,}8 \times s_R$)

—: more than 50 % of the raw data below the quantification limit, so results not treated

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Ethiopian Standards Agency

DRAFT

Organization and Objectives

The Ethiopian Standards Agency (ESA) is the national standards body of Ethiopia established in 2010 based on regulation No. 193/2010.ESA is established due to the restructuring of Quality and Standards Authority of Ethiopia (QSAE) which was established in 1970.

ESA's objectives are:-

- Develop Ethiopian standards and establish a system that enable to check weather goods and services are in compliance with the required standards,
- Facilitate the country's technology transfer through the use of standards,
- Develop national standards for local products and services so as to make them competitive in the international market.

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The Ethiopian Standards are developed by national technical committees which are composed of different stakeholders consisting of educational Institutions, research institutes, government organizations, certification, inspection, and testing organizations, regulatory bodies, consumer association etc. The requirements and/ or recommendations contained in Ethiopian Standards are consensus based that reflects the interest of the TC representatives and also of comments received from the public and other sources. Ethiopian Standards are approved by the National Standardization Council and are kept under continuous review after publication and updated regularly to take account of latest scientific and technological changes.
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